

FORMS PTO-1390
(REV. 10-90)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

Ball 1-2-3 09/530342

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

60/063,825

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

INTERNATIONAL APPLICATION NO.

PCT/US98/21872

INTERNATIONAL FILING DATE

15 October, 1998

PRIORITY DATE CLAIMED

31 October, 1997

TITLE OF INVENTION

APPARATUS AND METHOD FOR DRAWING WAVEGUIDE FIBERS

APPLICANT(S) FOR DO/EO/US

Corning Incorporated

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☐ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ has been transmitted by the International Bureau.
 - c. ☒ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☒ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. To 16. Below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An Assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

U.S. APPLICATION NO. (If known, rec 37 CFR 1.5)
60/063,825

09/530342

INTERNATIONAL APPLICATION NO.
PCT/US 98/21872ATTORNEY'S DOCKET NUMBER
Ball 1-2-317. ☒ The following fees are submitted:**BASIC NATIONAL FEE (37 CFR 1.492 (a)(1)-(5):**

Search Report has been prepared by the EPO or JPO.....\$840.00
 International preliminary examination fee paid to USPTO (37 CFR 1.482).....\$670.00
 No international preliminary examination fee paid to USPTO (37 CFR 1.482) but
 international search fee paid to USPTO (37 CFR 1.445(a)(2)).....\$760.00
 Neither international preliminary examination fee (37 CFR 1.482) nor international search
 fee (37 CFR 1.445(a)(2)) paid to USPTO.....\$970.00
 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims
 satisfied provisions of PCT Article 33(2)-(4).....\$96.00

CALCULATIONS

PTO USE ONLY

ENTER APPROPRIATE BASIC FEE AMOUNT = \$ 96.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from
 the earliest claimed priority date (37 CFR 1.492(e)).

\$

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
Total claims	- 20 =		X \$18.00	\$
Independent claims	- 3 =		X \$78.00	\$
MULTIPLE DEPENDANT CLAIM(S) (if applicable)			+ \$260.00	\$

TOTAL OF ABOVE CALCULATIONS = \$ 96.00

Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity
 Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28)

\$

SUBTOTAL = \$ 96.00

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30
 months from the earliest claimed priority date (37 CFR 1.492(f)).

\$

TOTAL NATIONAL FEE = \$

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
 accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property

\$ 40.00

TOTAL FEES ENCLOSED = \$ 136.00

Amount to be
 refunded:
 Charged: \$ 136.00

- a. ☐ A check in the amount of \$ _____ to cover the above fees is enclosed.
 b. ☒ Corning Incorporated hereby authorizes use of Deposit Account No. 03-3325 in the amount of
 \$ 136.00 to cover the above fees. A duplicate copy of this sheet is enclosed.
 c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or
 credit any overpayment to Deposit Account No. 03-3325. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

Send all correspondence to:

Randall S. Wayland
 Corning Incorporated
 SP-TI-03
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Signature

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Dated: 4-27-00

1/PRJ

09/530342
527 Rec'd PCT/PTO 27 APR 2000

Ball 1-2-3

APPARATUS AND METHOD FOR DRAWING WAVEGUIDE FIBERS

FIELD OF THE INVENTION

5 The present invention relates to a method and
apparatus for drawing waveguide fibers. More
particularly, the present invention relates to a furnace
that significantly reduces point defect losses in fibers
generated during the draw process.

10

BACKGROUND OF THE INVENTION

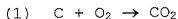
15 Relatively high temperature heat sources are required
for drawing high strength, low loss fibers from a high
silica-content fiber preform or blank. The two
predominant heat sources that have been utilized for
drawing such fibers are zirconia and graphite furnaces.
Fiber draw furnaces generally operate at temperatures
20 greater than about 1900°C, typically as high as about
2050°C.

25 A zirconia induction furnace conventionally includes
a housing in which there is a centrally disposed tubular,
yttria-stabilized zirconia susceptor surrounded by a
cylindrical quartz beaker containing granular zirconia
insulating material. An induction coil surrounding the
insulating material provides an alternating

electromagnetic field when energized. The field couples to the susceptor and raises the temperature of the susceptor to form a hot zone. An end portion of glass optical fiber preform is lowered into the hot zone to melt the end portion and a fiber is drawn from this melted end portion.

One disadvantage associated with zirconia induction furnaces is that extended use and thermomechanical stresses cause cracks in the muffle and susceptor. This cracking causes zirconia particles to migrate from the inner surface of the furnace onto the preform and/or fiber being drawn from the preform resulting in substantially weakened fiber and unacceptable product losses.

Graphite induction furnaces typically have a graphite muffle that is less susceptible to cracking, but graphite furnaces suffer from the disadvantage that the graphite muffle oxidizes at high drawing temperatures. It has been suggested that drawing a waveguide fiber in a graphite furnace must be performed in an inert protective atmosphere to prevent oxidation of the furnace muffle. Oxidation occurs when gasses from ambient atmosphere react with the solid carbon muffle at high temperatures according to the following reactions:



A typical onset temperature for reaction (1) for a graphite grade used in a draw furnace is about 700°C. Reaction (2) becomes significant above 900°C. These reactions of the furnace muffle with oxygen and carbon dioxide cause the furnace muffle to be consumed, especially at elevated fiber drawing temperatures.

The graphite muffle material is a composite of graphite grains bonded together by a carbon binder matrix. It is believed that the binder material is more

susceptible to oxidation than the graphite grains.

Therefore, when the composite of the two materials is exposed to oxygen at temperatures above the oxidation onset temperatures, the matrix binder material

preferentially oxidizes. The graphite grains, having no binder left to hold them place, are then free to fall away from the composite structure. It is believed that this mechanism causes graphite particulate to migrate from the muffle wall to the fiber preform and/or fiber during drawing.

Graphite particulate that becomes incorporated into the fiber during drawing causes unacceptable product losses due to point defects. Point defects manifest themselves as sharp attenuation increases in the signal transmitted through the fiber. Point defect product losses due to graphite particulate from a draw furnace losses can be greater than about 5%, which is an unacceptably high loss. Graphite particulate that has adhered to the fiber during the draw process also contributes to fiber breaks.

As mentioned above, it has been suggested that oxidation of the graphite furnace muffle may be overcome by drawing in an inert, protective gas atmosphere. The outer surface of a graphite muffle may be insulated by enclosing the muffle in a housing and flowing inert gas between the housing and the outer wall of the muffle. However, it is difficult to eliminate all oxygen from the furnace muffle, especially the inner surface of the muffle which is exposed to oxygen from ambient air that may leak into the furnace during loading and unloading waveguide fiber preforms. In addition, oxygen is believed to be present in the furnace due to the difficulty in eliminating oxidants from the furnace. For example, the upper region of the muffle is susceptible to oxidation

from the oxygen-containing porous soot section of an optical fiber blank that dwells in the furnace muffle during loading of the blank in the furnace. It is believed that oxygen present in the porous region of the blank oxidizes the muffle, producing graphite particulate.

In view of the above considerations, it would be desirable to provide a graphite fiber draw furnace muffle that does not generate graphite particulate, and thus significantly reduces point defect losses in the fiber.

SUMMARY OF INVENTION

Accordingly, the present invention generally provides an apparatus for heating a glass waveguide fiber preform to a temperature sufficient to draw a fiber therefrom comprising a generally tubular graphite muffle including an inner surface having a coating of high purity silicon carbide on the inner surface of the muffle. The coating preferably has a thickness of at least about 2 mils and contains less than about 900 parts per billion impurities.

In another aspect, the invention provides a method for producing a waveguide fiber in a draw furnace including a generally tubular graphite muffle having an inner surface. The method includes the steps of providing a high purity silicon carbon coating on the inner surface of the graphite muffle. The method further includes disposing a waveguide fiber preform in the furnace muffle, heating the furnace to a temperature sufficient to draw fiber from the preform, and drawing fiber from the blank.

Several important advantages will be appreciated from the foregoing summary. The principal advantage of the present invention is significantly reducing point defect losses in waveguide fibers drawn in a furnace having a graphite muffle. Additional features and advantages of

the invention will be set forth in the description which follows. It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory and are intended to provide further explanation of the invention as claimed. Various elements of the accompanying drawing are not intended to be drawn to scale, but instead are sometimes purposely distorted for the purposes of illustrating the invention.

BRIEF DESCRIPTION OF THE DRAWING

Fig. 1 is a schematic illustration of an exemplary embodiment of optical fiber draw furnace of the present invention.

DETAILED DESCRIPTION

Reference will now be made in detail to the present preferred embodiment of the invention, an example of which is illustrated in the accompanying drawing.

The present invention includes an apparatus for heating a waveguide fiber to a temperature sufficient to draw a fiber therefrom. An exemplary embodiment of the present invention is shown in Fig. 1 and is designated generally by reference numeral 10.

As embodied herein and referring to Fig. 1, furnace 10 is comprised of a generally cylindrical housing 12 having a side wall 14, a top portion 16, and a bottom portion 18. Top portion 16 has a central opening 22 therein which is vertically aligned with an opening 24 in bottom portion 18. Insulating material 26 is axially disposed in housing 12, which may be formed from a plurality of segments. A generally tubular, graphite

muffle 28 is centrally located within the insulating material 26. The muffle 28 and insulating material may be separated from the bottom portion 18 by a spacer ring 20 having an aperture 21 through which fiber is drawn to insulate the muffle from the bottom portion. The spacer ring 20 may be made from silica. An induction coil 30, which is connected to a power source (not shown), surrounds the insulating material 26 to provide a heating source for the furnace 10.

Housing 12, which is water cooled, may be fabricated of stainless steel or the like. Preferably, housing 12 axially runs the full length of the muffle 26 to fully enclose the muffle. An inert gas such as argon is flowed into the housing 12 to prevent oxidation of the outer surface of the muffle 26.

A waveguide fiber preform 32 (shown in phantom) is axially inserted into muffle 26 until a first end 34 thereof is positioned at the "hot zone" located within the induction coil 30. After hot zone has reached a temperature sufficient to draw fiber from the preform, which is preferably above 1900°C, an optical fiber 36 is drawn from the end portion 34 of the preform 32. In an important aspect of the invention, the inner surface of the muffle 28 adjacent the preform 32 has a coating of high purity silicon carbide thereon to prevent deterioration of the graphite muffle. The graphite muffle 28 preferably comprises at least two and, more preferably, three axial segments because it is difficult to coat sections of the muffle longer than about 40 inches.

The thickness of the silicon carbide coating is preferably at least about 2 mils and less than about 100 mils. Coating thinner than about 2 mils does not adequately prevent graphite particulate from contaminating fiber drawn from the furnace, and coating thicker than

about 100 mils is susceptible to microcracking and thermal shock. The thermal expansion of the SiC coating must be closely matched to the carbon binder matrix material which holds the graphite grains of the muffle together to prevent delamination of the coating due to thermal expansion mismatch.

The silicon carbide coating in the inner surface of the muffle is preferably formed by a chemical vapor deposition process using a silicon containing gas. Such a coating may be formed by reacting a silicon containing gas such as a silane with hydrogen to form SiC, wherein the silicon and carbon are present in a ratio of about one to one. The SiC is coated on the inner surface of the substrate which has been heated above 1000°C. High purity coatings are preferred on the inner surface of the draw furnace muffle to prevent contamination of fibers drawn in the furnace of the present invention. Preferably the impurity level in the silicon carbide coating is less than about 900 parts per billion, and more preferably less than about 200 parts per billion.

Another aspect of the present invention is directed to a method for producing a waveguide fiber in a draw furnace including a graphite, generally tubular muffle having an inner surface. The method comprises the steps of providing a high purity silicon carbide coating on the inner surface of the graphite muffle, disposing a waveguide fiber preform in the muffle, heating the furnace to a temperature sufficient to form draw fiber from the preform, and drawing fiber from the preform.

The furnace is preferably heated to a temperature of at least about 1900°C, more preferably to at least about 2000°C, to enable the tip of the waveguide preform to soften and allow fiber to be drawn therefrom. The high purity silicon carbide is preferably about 99.999% pure,

and more preferably contains less than about 900 parts per billion of impurities. The low impurity level is an important aspect of the present invention because higher impurity levels may cause optical or mechanical defects in the fiber produced in the furnace.

Waveguide fibers produced by utilizing the furnace and method of the present invention exhibit significantly reduced point defect losses. Fibers drawn in a conventional graphite muffle draw furnace exhibited product losses from attenuation due to point defects of approximately 5%. Fibers produced in a furnace of the present invention including a generally tubular, graphite muffle having an inner surface thereof coated with a silicon carbide layer about 5-8 microns thick exhibited product losses from attenuation due to point defects of approximately 0.8%.

It will be apparent to those skilled in the art that various modifications and variations can be made in the method and apparatus of the present invention without departing from the spirit or scope of the invention. Thus, it is intended that the present invention cover the modifications and variations of this invention provided they come within the scope of the appended claims and their equivalents.

CLAIMS

What is claimed is:

- 5 1. A furnace for heating a glass waveguide fiber preform to a temperature sufficient to draw a fiber therefrom comprising a graphite, generally tubular muffle including an inner surface having a coating of high purity silicon carbide on the inner surface of the muffle.
- 10 2. The furnace of claim 1, wherein the muffle further comprises at least two generally tubular sections.
- 15 3. The furnace of claim 2, wherein the muffle comprises three generally tubular sections.
4. The furnace of claim 1, wherein the coating has a thickness of at least about 2 mils.
- 20 5. The furnace of claim 1, wherein the silicon carbide contains less than about 900 parts per billion of impurities.
- 25 6. A method for producing a waveguide fiber in a draw furnace including a graphite, generally tubular muffle having an inner surface comprising the steps of:
 providing a high purity silicon carbide coating on the inner surface of the graphite muffle;
 disposing waveguide fiber preform in the muffle;
30 heating the furnace to a temperature sufficient to draw fiber from the preform; and
 drawing fiber from the preform.

7. The method of claim 6, wherein the temperature of furnace is at least about 1900°C.

5 8. The method of claim 6, wherein the temperature of the furnace is at least about 2000°C.

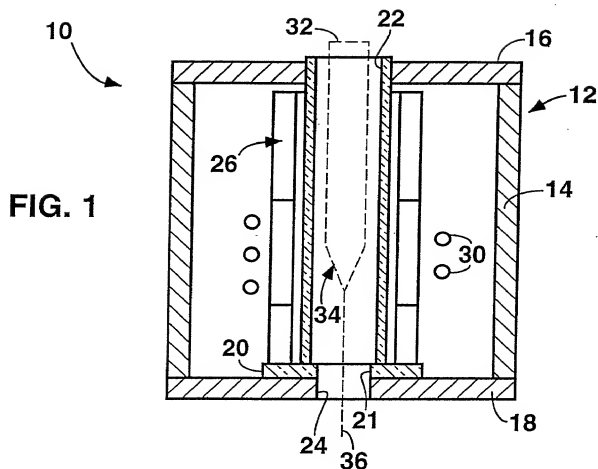
10 9. The method of claim 6, wherein the silicon carbide contains less than about 900 parts per billion of impurities.

11. The method of claim 6, wherein the waveguide fiber drawn from the furnace has a point defect loss less than about 4%.

15 11. The method of claim 1, wherein the waveguide fiber drawn from the furnace has a point defect loss less than about 1%.

ABSTRACT OF THE DISCLOSURE

A method and apparatus for drawing a waveguide fiber including a furnace having a graphite muffle with a coating of silicon carbide on the inner surface thereof. Fibers drawn in the furnace of the present invention exhibit greatly reduced point defect losses due to graphite particulate when compared to fibers drawn in graphite furnace muffles not having the silicon carbide coating.



DECLARATION IN ORIGINAL APPLICATION Attorney Docket No.: Ball 1-2-3

As a below named inventor, I declare that:

My residence, Post Office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled **APPARATUS AND METHOD FOR DRAWING WAVEGUIDE FIBERS**, the specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate, on the same subject matter, having a filing date before that of the application on which priority is claimed:

<input type="checkbox"/>	Country:	Application No.:	Filing Date:
<input checked="" type="checkbox"/>	PCT	PCT/US98/21872	Filing Date: 10/15/98

I hereby claim the benefit under Title 35 United States Code § 119(e) and § 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35 United States Code § 112, I acknowledge the duty to disclose material information as defined in Title 37 Code of Federal Regulations, § 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

<input checked="" type="checkbox"/>	Application Serial No.: 60/063825	Filed: 10/31/97	Status: Abandoned
<input type="checkbox"/>	NONE		

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

DECLARATION IN ORIGINAL APPLICATION

Attorney Docket No. Ball 1-2-3

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